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Volume III	Ethylene Dichloride	Volume X	Ortho-Xylene
Volume IV	Benzene	Volume XI	Maleic Anhydride
Volume V	Acetone	Volume XII	Dimethyl Terephthalate
Volume VI	Acrylonitrile	Volume XIII	Adipic Acid
Volume VII	Cyclohexanone	Volume XIV	Phthalic Anhydride

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ASSESSMENT OF CYCLOHEXANONE AS A POTENTIAL AIR POLLUTION PROBLEM

Volume VII

by

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GCA/TECHNOLOGY DIVISION
Bedford, Massachusetts

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SECTION I

SUMMARY AND CONCLUSIONS

Cyclohexanone is a colorless, slightly volatile liquid with an odor similar to acetone and peppermint. It is chemically stable and is manufactured mainly by catalytic dehydration of cyclohexanol. It is used extensively in the production of nylon and adipic acid, and it is also used as a solvent and degreaser. Cyclohexanone is a strong irritant and a narcotic agent at high concentrations, although concentrations producing such effects are unlikely to occur due to the low volatility of cyclohexanone. The occupational standard for an 8-hour time weighted exposure is 50 ppm.

Simple diffusion modeling estimates place the likely maximum 1-hour average ambient concentration at 1.3 ppm. The maximum 24-hour average ambient concentration might be expected to be about 1 ppm.

Approximately 850 million pounds of cyclohexanone were produced at 10 plants in 1974, with about 43 percent of this being used to make caprolactum for nylon 6, and 52 percent to make adipic acid. The remaining 5 percent was used as a solvent and as a degreasing agent. Total production is expected to increase at 10 percent per year for the next several years. Emissions result mainly from solvent usage, production losses, and bulk storage. About 6 percent of total production is eventually lost to the atmosphere.

Although emission controls specifically for cyclohexanone are not reported, two types of controls are used extensively by the chemical

industry to control hydrocarbon emissions. These are vapor recovery and incineration. Control by adsorption on activated charcoal is used when recovery is economically desirable. The primary advantage of incineration is that low concentrations may be oxidized with only small supplemental fuel requirements. Fixed roof storage tanks can be controlled by venting to an adsorber or to an incinerator, or they can be converted to floating roof design.

Based on the results of the health effects research presented in this report, and the ambient concentration estimates, it appears that cyclohexanone as an air pollutant does not pose a threat to the health of the general population. In addition, cyclohexanone does not appear to pose other environmental insults which would warrant further investigation or restriction of its use at the present time.

SECTION II

AIR POLLUTION ASSESSMENT REPORT

PHYSICAL AND CHEMICAL PROPERTIES

Cyclohexanone is a colorless to pale yellow, slightly volatile liquid with a ketone-type odor similar to acetone and peppermint. It is chemically stable and not very flammable except at high temperatures. The main method of manufacture is by the catalytic dehydration of cyclohexanol. Its most important use is as an intermediate in the manufacture of nylon 6, and it is also used as a metal degreaser, a solvent and a thinner for lacquers and synthetic resins. It is found in paint removers, and it is an excellent solvent for DDT, some organic phosphorous insecticides, and other similar materials.[1,2] Significant characteristics of cyclohexanone are listed in Table 1.

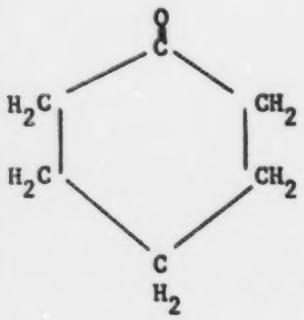
HEALTH AND WELFARE EFFECTS

Effects on Man

Acute Poisoning - Cyclohexanone is a strong irritant and a narcotic agent. Humans exposed for 3 to 5 minutes found 50 ppm irritating to the eyes, nose, and throat.[3] Exposure to higher levels and human response have not been documented in the literature, but animal studies indicate that human exposure to elevated levels would cause narcosis, dizziness, unconsciousness, and death due to respiratory failure. Concentrations producing such effects are unlikely to be encountered due to cyclohexanone's low volatility, except when handled at high temperatures.

Table 1. SIGNIFICANT PROPERTIES OF CYCLOHEXANONE[4]

Synonyms: anone, hexanon, hytrol o, ketohexamethylene, nadone, pimelic ketone, sextone

Chemical formula	
Molecular weight	98.14
Boiling point	155.6°C
Melting point	-45°C
Specific gravity	0.9478 at 20°/4°C
Vapor density	1.01 (air = 1)
Vapor pressure	5.2 mm Hg at 25°C
Solubility	2.4 g/100 ml water at 31°C, soluble in alcohols, ketones, esters, halogenated hydrocarbons. Partially soluble in benzene.
Lower explosive limit	1.1% by volume
Ignition temperature	420°C
Flash point	43.6°C (closed cup)
At 25°C and 760 mm Hg	1 ppm = 4.01 mg/m^3 1 mg/m^3 = 0.25 ppm

Its vapors have such strong warning properties at low concentrations that the acute exposure necessary to cause severe poisoning will not be tolerated voluntarily by humans. Irritation of the eyes, nose, and throat due to exposure at low concentrations is only temporary, with recovery after removal of the vapor.

Cyclohexanone has a low acute oral toxicity,[2] and absorption through the skin will not be a problem unless there is excessive exposure. It is capable of defatting the skin, and exposure to high concentrations may cause skin irritation.

Chronic Poisoning - Prolonged or repeated exposure will cause dermatitis.[5] It has been reported that no ill effects, except drowsiness, were observed in workers in daily contact with cyclohexanone.[2] No fatality or serious poisoning has been reported in the literature. Chronic exposure to concentrations able to produce delayed narcotic symptoms and death as shown in animal studies are not likely to be encountered in humans due to human sensory warning response at low levels. A concentration of 25 ppm has been estimated to be the highest concentration tolerable for an 8-hour exposure with no ill effects,[3] but the United States occupational standard for an 8-hour time weighted exposure average is 50 ppm.

Effects on Animals

Acute Poisoning - Studies done on animals illustrate the acute narcotic action of cyclohexanone. Table 2 summarizes available dose-response data from various inhalation studies.[1,6,7] Symptoms in guinea pigs prior to death were narcosis; lachrymation; excess salivation; depression of body temperature, respiratory rate, and heart rate; and opacity of the cornea. Recovery was slow, with some guinea pigs dying within 4 days.[1] A concentration of 2,000 ppm inhaled over 4 hours is the lowest recorded concentration able to cause death in susceptible rats.[7]

Table 2. ACUTE EFFECT OF CYCLOHEXANONE ON ANIMALS

Animal	Dose, ppm	Time, hr	Response	Reference
Rat	8,000	4	Anesthesia and death	6
Guinea pig	4,000	6	Narcosis; death to some within 4 days	1
Rat	4,000	4	Narcosis; all survived	6
Rat	2,000	4	Narcosis; some deaths	7

Rabbits were killed by the absorption of 10.2 to 23.0 g/kg body weight through clipped uncovered skin.[8] Symptoms prior to death were marked hypothermia, convulsions, and narcosis. Pure cyclohexanone dropped into the eyes of rabbits caused irritation and corneal injury.[9] Oral administration of 1600 mg/kg body weight to rabbits[8] resulted in narcosis and death within a day. An LD_{50} value of 1620 mg/kg body weight has been given for rats.[7]

Chronic Poisoning - Monkeys and rabbits were exposed for 6 hours per day, 5 days per week, for 10 weeks to various concentrations of cyclohexanone.[10] At 190 ppm there were no detectable effects or abnormal behavior except very slight liver and kidney injury. At 309 ppm, there was slight eye irritation. At 773 ppm, salivation increased as did eye irritation. Death occurred among animals exposed to 3082 ppm after 3 weeks of the exposure periods. Prior to death the animals became lethargic, showed loss of coordination, secreted mucus, and entered a narcotic state. No hematological disorders could be linked to cyclohexanone exposure.

Effects on Vegetation

The effects of cyclohexanone on plants have not been documented in the literature. However, due to its properties as a solvent and as a

defatting agent, it would act as an acute contact poison on plant parts, especially the leaves.

Other Effects

Cyclohexanone can be used as a sole carbon source for growth by a species of bacteria.[11] Due to its properties as a solvent, it could attack and dissolve some rubbers and plastics.

AMBIENT CONCENTRATIONS AND MEASUREMENT

Ambient Concentration Estimates

Although cyclohexanone emissions are greatest from the solvent usage source category, these sources tend to be small and geographically scattered. Production of cyclohexanone, however, occurs at a few locations for which the emissions characteristics can be fairly well defined, and which as single point or area sources have a large emission density.

The largest installation for cyclohexanone production is located in a town of about 1,800 population, and it has a capacity of about 240 million lb/yr. Assuming a 1 percent loss, this converts to an emission rate of:

$$\frac{(0.01 \text{ emission factor})\ (240 \times 10^6 \text{ lb/yr})\ (453.6 \text{ g/lb})}{3.1536 \times 10^7 \text{ sec/yr}}$$

= 34.5 g/sec of cyclohexanone.

Some assumptions must be made regarding this release to the atmosphere. First of all, the emissions do not all come from one source location, but rather from a number of locations within the plant where vapor leaks to the atmosphere. Thus, the emissions can be characterized as coming

from an area source which will be taken to be 100 meters on a side. Secondly, the emissions occur at different heights, and an average emission height of 10 meters is assumed.

Ground level concentrations can then be estimated at locations downwind of the facility.[12] To do this a virtual point source of emission is assumed upwind of the facility at a distance where the initial horizontal dispersion coefficient equals the length of a side of the area divided by 4.3. In this case:

$$\sigma_{yo} = 100\text{m}/4.3 = 23.3\text{m} .$$

Assuming neutral stability conditions (Pasquill-Gifford Stability Class D) with overcast skies and light winds, the upwind distance of the virtual point source is approximately 310 meters. With consideration of the plant boundary, it is reasonable to assume that the nearest receptor location is thus about 500 meters from the virtual point source. Finally, taking 2 m/sec as an average wind speed, the ground level concentration may be calculated from:

$$\chi = \frac{Q}{u\pi\sigma_y\sigma_z} e^{-\frac{1}{2}\left(\frac{H}{\sigma_z}\right)^2}$$

or

$$\chi = \frac{34.5}{(2)\ \pi\ (36)\ (18.5)} e^{-\frac{1}{2}\left(\frac{10}{18.5}\right)^2}$$

$$= 7.124 \times 10^{-3}\ \text{g/m}^3$$

for a 10-minute average concentration. Over a period of an hour this becomes $(7.124 \times 10^{-3}\ \text{g/m}^3)\ (0.72) = 5.129 \times 10^{-3}\ \text{g/m}^3$ or 1.3 ppm 1-hour average concentration. Over a 24-hour period, the average concentration might roughly be expected to be about 1.0 ppm.

Measurement Techniques

Measuring cyclohexanone in air involves its adsorption on charcoal with subsequent desorption and analysis by a gas chromatograph.[13]

In this method a known volume of air is drawn through a charcoal tube on which the organic vapors are adsorbed. The tube is then transferred to a small stoppered container where it is desorbed with carbon disulfide. An aliquot of the desorbed sample is injected into a gas chromatograph. The area of the resulting peak is determined and compared with areas obtained from the injection of standards. Concentrations in the range of 2.5 to 125 ppm can be readily detected by this method. Interferences will result if the amount of water in the air is so great that condensation in the tube will affect the collection efficiency, and other interferences will result from compounds having similar retention times. This technique is especially well suited for air pollution work, since there is no requirement for special chemicals in the field.

SOURCES OF CYCLOHEXANONE EMISSIONS

Cyclohexanone Production and Consumption

The production of cyclohexanone in 1974 was approximately 850 million pounds, and it is expected to increase at 10 percent per year for the next several years.[14] Presently, about 43 percent of all cyclohexanone is used to make caprolactam for nylon 6 and 52 percent is oxidized as mixed oil (cyclohexanone and cyclohexanol) to make adipic acid. The remaining 5 percent is used as a solvent. Nine companies at ten locations are producing both cyclohexanone and cyclohexanol. The names and locations of production facilities are given in Appendix A.

The consumption of cyclohexanone for final products is shown in Table 3.

Table 3. CYCLOHEXANONE CONSUMPTION - 1974[14,15]

Product	Million pounds	% Annual growth
Adipic acid	442	5.5
Caprolactam	365.5	17.0
Solvent	42.5	5.0

Cyclohexanone Sources and Emission Estimates

Primary sources of emissions of cyclohexanone result from solvent usage, production losses, and bulk storage. Total emissions from all categories in 1974 are estimated to have been 51.3 million pounds as shown in Table 4, representing 6 percent of total production.

Table 4. SOURCES AND EMISSION ESTIMATES OF CYCLOHEXANONE - 1974

Source	Million pounds
Solvent usage	42.5
Production losses	8.5
Storage	0.3
Total	51.3

The major source of cyclohexanone emissions results from its use as a solvent. It is used in the manufacture of lacquers and crude rubber, as a spot remover, and as a degreaser for leather. Since 1967, sales have increased due to its use as a solvent for coatings, especially as a replacement for isophorone in vinyl solution coatings. This replacement is due mainly to avoid new solvent regulations, since cyclohexanone is

considered a nonreactive solvent. However, a recent EPA report has classified it as highly reactive.[16] Assuming that all cyclohexanone used as a solvent will evaporate to the atmosphere, 42.5 million pounds of cyclohexanone are emitted due to solvent usage.

The second major source of emissions occurs from the production of cyclohexanone. Most cyclohexanone and cyclohexanol is prepared as a mixture by the catalytic oxidation of cyclohexane. If only cyclohexanone is desired, the cyclohexanone/cyclohexanol mixture can be dehydrogenated with a zinc oxide catalyst to give an essentially pure product. Cyclohexanone may also be produced by the hydrogenation of phenol using a palladium on carbon catalyst. Palladium emissions may result from the process operation and from the disposal of spent bed material.

Since data are not available concerning emissions from these processes, based on other similar chemical processes,[17,18] it is estimated that 1 percent of production is emitted as cyclohexanone resulting in 8.5 million pounds of emissions.

The last major source of emissions results from bulk storage of cyclohexanone. Using emission factors in AP-42[17] and assuming all storage tanks are fixed roof, emissions are 0.3 million pounds.

CYCLOHEXANONE EMISSION CONTROL METHODS

The literature does not report specific control equipment for cyclohexanone, but it does report on control devices for other similar hydrocarbons. Two types of control devices are presently used by the industry to control hydrocarbon emissions: vapor recovery and incineration. Both systems have reported efficiencies of at least 95 percent.

Control of hydrocarbon emissions by adsorption on activated charcoal is generally applied when recovery of adsorbed material is economically

desirable. Because of the heat generated in adsorbing ketones, the temperature of the bed must be cooled by adding moisture to the gas stream. Adsorption is generally used when concentrations of hydrocarbons are greater than 2500 ppm.[19] Other applications are for the control of very low concentration hydrocarbons that are poisonous to catalytic incinerators, and for collection and concentration of low concentration emissions for subsequent disposal by incineration. Cost data for the cases utilizing adsorption are presented in Tables 5 and 6. The three cases presented are adsorption with solvent recovery, adsorption with incineration, and adsorption with incineration plus heat recovery.

Table 5. ESTIMATED INSTALLED COSTS[a] OF ADSORPTION SYSTEMS[20]

Adsorber capacity, SCFM - based on 25 percent lower explosive limit	1,000	10,000	20,000
With solvent recovery, $	74,000	162,300	280,000
With thermal incineration/ no heat recovery, $	89,500	202,000	344,000
With thermal incineration/ primary heat recovery, $	101,500	255,000	431,000

[a]Costs updated to first quarter 1975.

Table 6. ESTIMATED ANNUAL OPERATING COSTS[a] OF ADSORPTION SYSTEMS[20]

Adsorber capacity, SCFM - based on 25 percent lower explosive limit	1,000	10,000	20,000
With solvent recovery, $/yr	13,200	10,479[b]	37,200[b]
With thermal incineration/ no heat recovery, $/yr	23,400	64,300	123,200
With thermal incineration/ primary heat recovery, $/yr	25,600	82,000	141,600

[a]Costs updated to first quarter 1975.

[b]Indicates a savings.

Control of cyclohexanone emissions by incineration or catalytic oxidation involves direct oxidation of the combustible portion of the effluent, the desired ultimate products being water and carbon dioxide.

The primary advantage of catalytic incineration is that extremely small concentrations of organics can be oxidized with only small amounts of supplemental fuel required. The main disadvantages are the higher capital cost and the fact that certain hydrocarbons may poison the catalyst. Cost data for thermal and catalytic incinerators with and without heat recovery are presented in Tables 7 and 8.[20]

Control of emissions from storage tanks will require the use of floating roof tanks or venting the emissions to the previously mentioned adsorber or incinerator. Emissions from fixed roof tanks can be vented to either system without any major increase in cost. If these systems are not available, the fixed roof tanks should be switched to floating roof tanks resulting in a 90 percent reduction of emissions. Figure 1 provides estimated costs of various gasoline storage tanks.[20] These equipment cost estimates can also be applied to cyclohexanone. As can be seen, conversion of fixed roof to floating roof tanks by installation of internal floating covers is more economical than the installation of new pontoon floating tanks.

Table 7. ESTIMATED INSTALLED COSTS[a] OF THERMAL AND CATALYTIC INCINERATORS[20]

Incinerator capacity, SCFM - based on 25 percent lower explosive limit	1,000	10,000	20,000
Installed costs, $			
Catalytic without heat recovery	43,500	272,000	504,600
Catalytic with primary heat recovery	54,100	306,000	573,900
Catalytic with primary and secondary heat recovery	68,300	361,800	666,400
Thermal without heat recovery	27,200	92,500	137,400
Thermal with primary heat recovery	40,300	144,200	232,600
Thermal with primary and secondary heat recovery	54,400	200,000	322,300

[a]Costs updated to first quarter 1975.

Table 8. ESTIMATED ANNUAL OPERATING COSTS[a] OF THERMAL AND CATALYTIC INCINERATORS[20]

Incinerator capacity, SCFM -- based on 25 percent lower explosive limit	1,000	10,000	20,000
Operating costs, $/yr			
Catalytic without heat recovery	16,200	102,800	195,000
Catalytic with primary heat recovery	16,400	78,500	177,900
Catalytic with primary and secondary heat recovery	19,300	108,700	203,700
Thermal without heat recovery	12,000	54,300	96,700
Thermal with primary heat recovery	11,500	36,300	59,200
Thermal with primary and secondary heat recovery	14,400	50,800	84,500

[a]Costs updated to first quarter 1975.

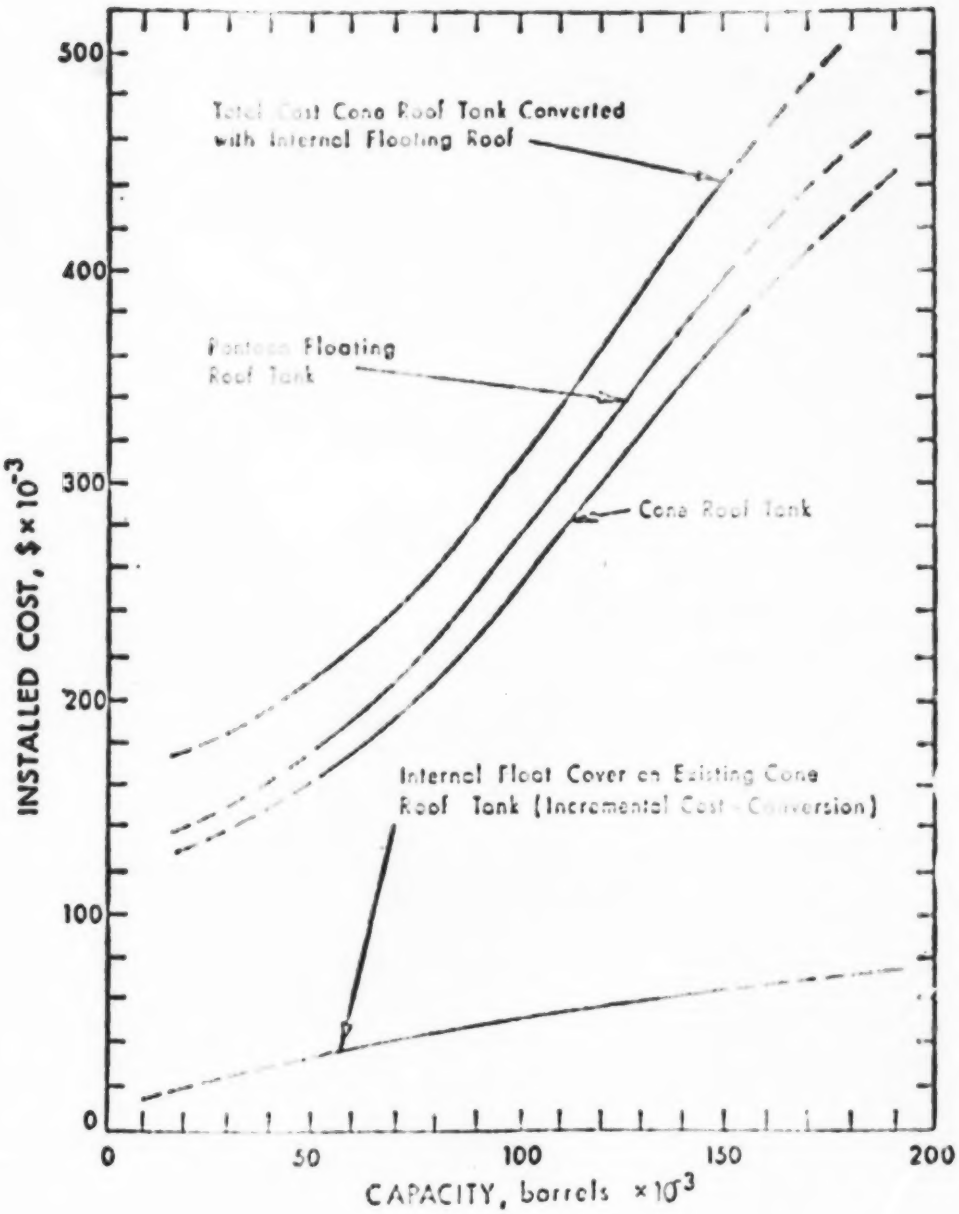


Figure 1. Estimated installed cost of cyclohexanone storage tanks (equipment costs assumed to be the same as gasoline storage tanks)[20]

SECTION III

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APPENDIX A

CYCLOHEXANONE MANUFACTURERS

		Estimated capacity, million lb/yr
Allied Chemical Corp.	Hopewell, Virginia	173
Celanese Corp.	Bay City, Texas	50
Dow Badische Co.	Freeport, Texas	125
DuPont	Belle, West Virginia	240
El Paso Natural Gas Co.	Odessa, Texas	32
Monsanto Co.	Luling, Louisiana	250
Monsanto Co.	Pensacola, Florida	
Nipro	Augusta, Georgia	75
Rohm and Haas Co.	Louisville, Kentucky	20
Union Carbide Corp.	Taft, Louisiana	35
	Total	1000